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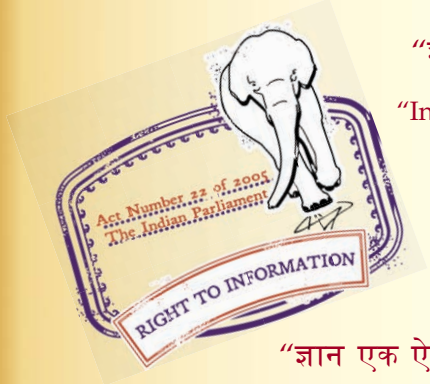
IS 5045 (1989): Dye Intermediates - Metanilic Acid, Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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"Knowledge is such a treasure which cannot be stolen"

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IS 5045 : 1989
(Reaffirmed 2000)

Indian Standard

DYE INTERMEDIATES — METANILIC ACID, TECHNICAL — SPECIFICATION

(Second Revision)

First Reprint SEPTEMBER 1997

UDC 547·551·52'022

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

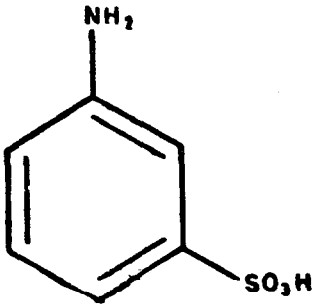
August 1990

Price Group 3

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards on 17 November 1989, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Metanilic acid ($C_6H_7O_3NS$), also described by such chemical names as *m*-aminobenzene sulphonic acid and aniline-3-sulphonic acid, is an important dye intermediate widely used in the manufacture of azo dyes, namely, metanil yellow (C. I. 13065), sulphocyanine 5R (C. I. 26400), and cotton black (C. I. 53140). It is also used in the manufacture of *m*-aminophenol and some optical whitening agents. It has the following structural formula:



Metanilic Acid
(Molecular Mass 173·2)
CAS Registry Number [121-47-1]

This standard was first issued in 1969 and revised in 1976 when the requirement for sulphanilic acid content and method for its determination was incorporated. The Committee responsible for the preparation of this standard decided to revise it again in order to update the requirement for sulphanilic acid and also to incorporate the requirements for orthanilic acid, aniline 2, 5-disulphonic acid and iron content along with the methods for their determination.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

DYE INTERMEDIATES — METANILIC ACID, TECHNICAL — SPECIFICATION

(Second Revision)

1 SCOPE

1.1 This standard prescribes the requirements and the methods and test for metanilic acid, technical.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
265 : 1976	Specification for hydrochloric acid (*second revision*)
1070 : 1977	Specification for water for general laboratory use (*second revision*)
2552 : 1979	Specification for steel drums (galvanized and ungalvanized) (*second revision*)
5299 : 1969	Methods of sampling and tests for dye intermediates

3 REQUIREMENTS

3.1 Description

The material shall be in the form of paste or, if dry, in the form of yellowish-white to pinkish crystalline powder. It shall be free from grit and other visible impurities.

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS 2552 : 1979) lined with suitable polyethylene film, or as agreed to between the purchaser and the supplier.

4.2 Marking

Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;

b) Indication of the source of manufacture;

c) Gross mass, tare and net mass;

d) Batch number; and

e) The minimum cautionary notice worded as under:

'IT IS A MILD SENSITIZER. LOCAL CONTACT MAY CAUSE DERMATITIS'

5 SAMPLING

5.1 Representative samples of the material shall be drawn as precribed in **3** of IS 5299 : 1969.

5.2 Number of Tests

5.2.1 Test for assay, sulphanilic acid, orthanilic acid and aniline 2, 5-disulphonic acid shall be conducted on each of the individual samples.

Table 1 Requirements for Metanilic Acid, Technical

(*Clauses* 3.2, 5.3.1, 5.3.2 *and* 6.1)

Sl No.	Characteristic	Requirement (on Dry Basis)	Method of Test, Ref to Cl No. in Annex A
(1)	(2)	(3)	(4)
i)	Assay, percent by mass, *Min*	98	A-2
ii)	Sulphanilic acid, percent by mass, *Max*	0·5	A-3
iii)	Orthanilic acid, percent by mass, *Max*	0·2	
iv)	Aniline 2, 5-disulphonic acid, percent by mass, *Max*	0·3	
v)	Matter insoluble in sodium carbonate solution, percent by mass, *Max*	0·30	A-4
vi)	Iron content, ppm, *Max*	100	A-5

5.2.2 Tests for determination of remaining characteristics, namely, description, iron content and matter insoluble in sodium carbonate solution shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirement of assay, sulphanilic acid, orthanilic acid and analine 2, 5-disulphonic acid if each of the individual test results satisfies the relevant requirements given in Table 1.

5.3.2 *For Composite Sample*

For declaring the conformity of the lot to the requirements of all other characteristics, namely, description, matter insoluble in sodium carbonate solution and iron content, tested on the composite sample, the test results for each of the characteristics shall satisfy the relevant requirements given in Table 1.

6 TEST METHODS

6.1 Tests shall be carried out according to the methods prescribed in Annex A. Reference to the relevant clauses of the Annex is given in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(*Table* 1, and *Clause* 6.1)

METHODS OF TEST FOR METANILIC ACID, TECHNICAL

A-1 PREPARED SAMPLE

A-1.1 Dry the material at 105 ± 1°C to constant mass. Grind and mix well. Transfer the material to a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this prepared sample for tests.

A-2 ASSAY

A-2.1 Outline of the Method

The material is titrated directly under acidic conditions with standard sodium nitrite solution using starch-iodide test paper. The method makes no distinction between metanilic acid and other amino compounds and determines amine content of the sample which is calculated as metanilic acid.

A-2.2 Reagents

A-2.2.1 *Sodium Carbonate Solution*, approximately 20 percent (*m/v*).

A-2.2.2 *Concentrated Hydrochloric Acid*, *see* IS 265 : 1976.

A-2.2.3 *Standard Sodium Nitrite Solution*, 0·2 N (freshly standardized).

A-2.2.4 *Starch-Iodide Test Papers*

A-2.3 Procedure

Weigh accurately about 17 g of the prepared sample (*see* A-1.1) and dissolve by heating in 100 ml of sodium carbonate solution. Cool and make up to volume with distilled water in a 1 000 ml volumetric flask. Pipette 50 ml aliquot of this solution in a beaker and add, while stirring, 25 ml of hydrochloric acid. Cool to 20°C by adding ice-cold water. While stirring mechanically, titrate the solution with standard sodium nitrite solution which is added through a thistle funnel, the end of which is well under the solution. Add sodium nitrite solution as rapidly as it is consumed. The end point is reached when a drop of the solution spotted on a starch-iodide test paper produce an immediate blue coloured ring which may be repeatedly obtained during next 10 minutes without further addition of sodium nitrite.

A-2.4 Calculation

Assay (calculated on the basis of total amine content and molecular mass 173·2), percent by mass $= \frac{346{\cdot}4 \times V \times N}{M}$

where

V = volume in ml of standard sodium nitrite solution used,

N = normality of standard sodium nitrite solution, and

M = mass in g of the prepared sample taken for the test.

A-3 DETERMINATION OF SULPHANILIC ACID, ORTHANILIC ACID AND ANILINE 2, 5-DISULPHONIC ACID

A-3.0 General

The impurities in metanilic acid are estimated by descending paper chromatographic method.

A-3.1 Apparatus

A-3.1.1 *Chromatographic Chamber* (approx 270 mm × 150 mm × 400 mm).

A-3.1.2 *Sprayer*

A-3.1.3 *Micropipette*, 10 µl capacity.

A-3.2 Reagents

A-3.2.1 *m*-Butanol

A-3.2.2 *Tert* — Butanol

A-3.2.3 *Ammonia Solution*

A-3.2.4 *Concentrated Hydrochloric Acid*

A-3.2.5 *Sulphanilic Acid*, pure.

A-3.2.6 *Orthanilic Acid*, pure.

A-3.2.7 *Aniline 2, 5-Disulphonic Acid*, pure.

A-3.2.8 *Developing Solvent, Tert* — Butanol : *N* butanol : (4 : 3 : 3).

A-3.2.9 *Spray Solution*

Weigh accurately 1·0 g of *p*-dimethylamino benzaldehyde and dissolve in 5 ml of concentrated hydrochloric acid. Add 95·0 ml of rectified spirit to make up to 100 ml. Mix well.

A-3.3 Method

A-3.3.1 *Preparation of Solutions*

a) *Sample Solution*

Weigh exactly 1·0 g (100 percent) of well mixed prepared sample. Dissolve in ammonia solution and make up to 100 ml with distilled water. Mix well. Spot 0·01 ml of this solution.

b) *Standard Solution of Aniline 2, 5-Disulphonic Acid*

Weigh 1·0 g (100 percent) of aniline—2, 5-disulphonic acid and dissolve it in ammonia solution. Make up to 100 ml in standard volumetric flask with distilled water. Mix well. Pipette 0·3 and 0·5 ml in two 100 ml standard volumetric flasks and make up to mark with distilled water. Mix well. Call these solutions *A* and *B*. Spotting of 0·01 ml of these solutions *A* and *B* will be equivalent to 0·3 and 0·5 percent of aniline-2, 5-disulphonic acid respectively.

c) *Standard Solution of Sulphanilic Acid*

Weigh 1·0 g (100 percent) of sulphanilic acid and dissolve it in ammonia solution. Make up to 100 ml in standard volumetric flask with distilled water. Mix well. Pipette 1·0 and 2·0 ml in two 100 ml standard volumetric flasks and make up to mark with distilled water. Mix well. Call these solutions *C* and *D*. Spotting of 0·01 ml of these solutions *C* and *D* will be equivalent to 1·0 and 2·0 percent of sulphanilic acid, respectively.

d) *Standard Solution of Orthanilic Acid*

Weigh 1·0 g (100 percent) of orthanilic acid and dissolve it in ammonia solution. Make up to 100 ml in standard volumetric flask with distilled water. Mix well. Pipette 0·2 ml in 100 ml standard volumetric flask and make up to mark with distilled water. Mix well. Call this solution *E*. Spotting of 0·01 ml of solution *E* will be equivalent to 0·2 percent of orthanilic acid.

A-3.3.2 *Preparation of Tank*

Take about 50 ml developing mixture in the beaker and place it at the bottom of the developing tank and replace the lid. This is necessary to carry out chromatography in an atmosphere already saturated with the vapour of the developing solvent.

A-3.3.3 *Development of Chromatogram*

Place a solvent trough in the position. After spotting, place the paper in the trough so that it is suspended from one end and the other end rests freely into the tank. Make sure the papers hang freely, not touching each other or the sides of the tank. Each trough can take two papers. Add solvent in the trough, replace the lid and leave the chromatogram to develop for 16 hours.

After developing and drying at 50°C, it is sprayed with 1 percent solution of *p*-dimethylamino benzaldehyde. The chromatogram shown in Fig. 1 develops.

A-3.4 Estimation

After the chromatogram is developed, compare with the standard and evaluate.

A-4 MATTER INSOLUBLE IN SODIUM CARBONATE SOLUTION

A-4.1 Reagent

A-4.1.1 *Sodium Carbonate Solution*, approximately 10 percent (*m*/*v*).

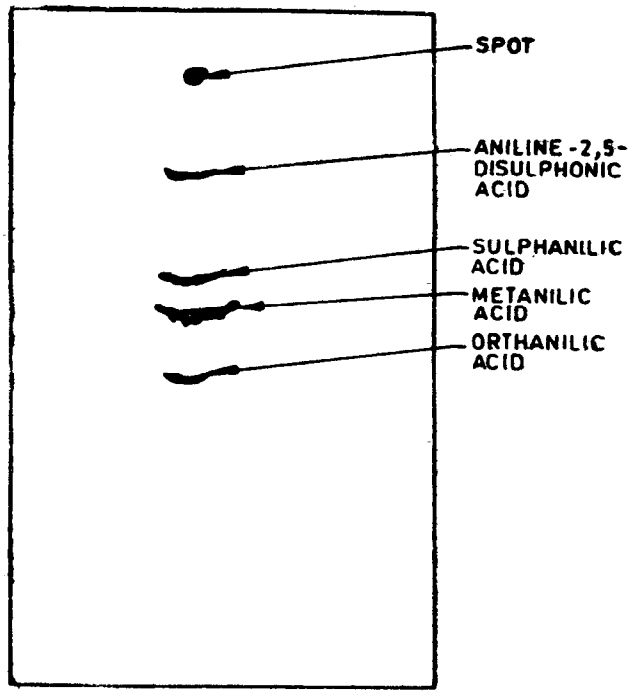


FIG. 1 TYPICAL CHROMATOGRAM IN ESTIMATION OF IMPURITIES IN METANILIC ACID

A-4.2 Procedure

Weigh accurately about 10·0 g of the prepared sample in 250-ml beaker. Add 150 ml water and 20-25 ml of 10 percent (*m/v*) sodium carbonate solution. Warm to 60°C while stirring to dissolve the material completely. Filter through a weighed sintered glass crucible of porosity G 4. Wash thoroughly with water. Dry at 100°C, cool to room temperature and weigh. Calculate the matter insoluble in sodium carbonate solution.

A-4.3 Calculation

$$\text{Matter insoluble in sodium carbonate solution, percent by mass} = \frac{M_1}{M} \times 100$$

where

M_1 = mass in g of the residue, and
M = mass in g of the material taken for the test.

A-5 DETERMINATION OF IRON CONTENT

A-5.1 Reagents

A-5.1.1 *Concentrated Hydrochloric Acid,* iron-free.

A-5.1.2 *Citric Acid,* iron-free, 20 percent (*m/v*).

A-5.1.3 *Thioglycolic Acid*

A-5.1.4 *Liquid Ammonia,* iron-free, 25 percent (*m/v*).

A-5.1.5 *Standard Iron Solution*

Dissolve 0·173 g of ferric ammonium sulphate with 1·5 ml of iron-free concentrated hydrochloric acid in a 1 000 ml flask and make up to mark with water. One millilitre of this solution contains 0·02 mg of iron.

A-5.2 Apparatus

A-5.2.1 *Muffle Furnace*

A-5.2.2 *Nessler Cylinders,* 50 ml (*see* IS 4161 : 1967).

A-5.2.3 *Platinum Dish*

A-5.2.4 *Water Bath*

A-5.3 Procedure

A-5.3.1 Weigh accurately 1 g of the sample in a clean and dry platinum dish and incinerate the material by an open flame carefully by gradually increasing the heat and then in a muffle furnace at about 800°C for about 2 to 4 hours until free

from carbon. Remove the platinum dish from the muffle furnace, cool and add a few millilitres of iron-free concentrated hydrochloric acid to the ash (**A-5.1.1**). Place the platinum dish on a boiling water bath until the hydrochloric acid evaporates to dryness Cool the platinum dish and moisten the residue with 5-10 drops of concentrated hydrochloric acid and 10 ml of water and heat further on the water bath for a few minutes. Allow the solution to cool and transfer to a 50-ml Nessler cylinder quantitatively. Wash the platinum dish with few millilitres of water and transfer the washings to the same Nessler cylinder and make volume approximately to 40 ml. Add 2 ml of 20 percent (*m/v*) iron-free citric acid solution to the Nessler cylinder followed by 2 to 5 drops of thioglycolic acid. Mix the solution with a glass rod and make alkaline with ammonia solution and dilute to 50 ml with water. Mix the solution and set aside for 5 to 10 minutes.

A-5.3.2 Simultaneously, in another Nessler cylinder, take 5·0 ml of standard iron solution (**A-5.1.5**) equivalent to 100 ppm of iron and develop the colour as above using the same amount of reagents. Dilute the solution to 50 ml with water and set aside for 5 to 10 minutes along with the sample.

The sample passes the test if the colour produced by the sample solution (**A-5.3.1**) is not deeper than that produced by the standard solution (**A-5.3.2**).

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